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EFFECT OF ADDITIVES ON THE CATALYTIC DECOMPOSITION
OF HYDROGEN PEROXIDE

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ABSTRACT

The influence of phosphate, tin, aluminum, sulfate, nitrate, chloride and ammonia on the decomposition of hydrogen peroxide with a silver catalyst have been studied. The first three of these alter the behavior of the peroxide considerably. All three increase the starting activity in a laboratory apparatus, aluminum being the most effective. Phosphate and tin exhibit inhibitory properties during the later phases of the decomposition runs and both increase the rate of silver solution. Phosphate has a greater influence than tin in both of these respects. The extent of starting activity, inhibition and catalyst loss is dependent on the concentration of these substances but they appear to reach their maximum effect at low concentrations. Some combinations of these three materials interact with each other but the subject has not been investigated sufficiently to allow the drawing of conclusions at present.

PROBLEM STATUS

This is an interim report; work on the problem is continuing.

AUTHORIZATION

NRL Problem C01-05
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EFFECT OF ADDITIVES ON THE CATALYTIC DECOMPOSITION OF HYDROGEN PEROXIDE

Introduction

The exothermic decomposition of hydrogen peroxide into oxygen and steam is the basis for the use of this material as a source of power. Although this decomposition can be initiated by thermal means, in practice it is accomplished by the use of catalysts. Silver is one of the most active catalysts and has been incorporated into several units or motors designed to deliver power or thrust from the decomposition of hydrogen peroxide. However, difficulties have been observed in the use of this noble metal element. The chief drawback experienced is that the life of the catalyst beds have been shorter than that anticipated. The undesirable behavior has manifested itself as a pressure build-up across the catalyst bed which decreases the flow rate of propellant. This pressure build-up is due to plugging of the beds in some cases but also has been observed in beds containing no debris. Where plugging has been found, stripping of silver from the top part of the bed and redeposition in lower regions is largely responsible for the trouble. Although some of the difficulty may be traced to the catalyst, there is good evidence that different peroxide samples exhibit a wide variation in behavior when used in the same type decomposition units.

Attempts have been made to relate the catalytic decomposition behavior of hydrogen peroxide samples to their physical properties such as pH and conductivity [reference (1)], tin and nitrate content [reference (1)], evaporation residues [reference (2)], ebullition temperatures [reference (3)], and to the solubility of silver in the peroxide [reference (3)]. None of these properties have been correlated conclusively with decomposition behavior and recent efforts have been directed toward the measurement of catalytic activity at low pressures under controlled conditions. A dynamic system has been developed and utilized by the Buffalo Electrochemical Co. which measures the activity of a 5/8 inch diameter, 20-mesh silver screen at a peroxide flow rate of 60 ml/min. [reference (4)]. At this Laboratory a static system has been adopted in which a stirred sample of peroxide is maintained at a constant temperature as it decomposes in the presence of a solid silver wire [reference (3)]. A 2.0 cm. length of 0.020 inch wire, which has been treated with dilute potassium cyanide to remove the tarnish, has been found to give satisfactory gas evolution rates with a 50 ml. sample. A control temperature of 60°C is normally used and the volume of oxygen formed in the decomposition is

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measured with a wet test meter. Periodic weighing of the wire in the course of a run permits the measurement of the silver solution rate.

With this laboratory set-up, different hydrogen peroxide samples gave greatly different decomposition activities and catalyst erosion rates. In two instances, samples showing high silver solution had given trouble in high pressure decomposition units [reference (3)] and it is probable that the solubility of silver in hydrogen peroxide is primarily responsible for the pressure build-up associated with plugged catalyst beds. There has been speculation that the pressure build-up observed with unclogged decomposition units is due to the activity of the peroxide, although no definite proof of this exists. Assuming these two reasons for the pressure build-up to be correct and with the knowledge that different peroxides do exhibit varying activities it is desirable to see what is responsible for the differences in decomposition behavior. In the work described below, two approaches have been used: (1) chemical analysis of production samples and (2) effect of additives on catalyst activity and solution rate.

Catalytic Decomposition of Production Samples of Hydrogen Peroxide

The decomposition behavior of hydrogen peroxide in the NRL static evaluator depends on the history and origin of the particular sample tested. This is well illustrated in Figure 5, where the gas evolution versus time curves for four hydrogen peroxide samples are shown. Although the maximum rate during a run was approximately the same for all four samples, the starting activity showed a ten-fold variation. All of the samples except HN increased in activity as the run proceeded before falling off further along in the reaction. Sample HN started at a high rate which was maintained for about 15 minutes before decreasing in activity, a behavior which approximates that which would be expected from all samples as the concentration of hydrogen peroxide falls off. The upturn in gas evolution rate for samples HN, LN and D-3 after about 40-50 minutes of operation was due to the precipitation of silver which had been dissolved from the catalyst by the initially more concentrated peroxide and which could be held in solution no longer as the peroxide strength decreased. Decomposition throughout the solution as well as on the wire surface can be observed when this occurs and the reaction must be terminated in order to prevent an uncontrollable, self-accelerating rate increase.

The re-precipitation of silver in any particular run depends on the extent of decomposition and the amount of silver dissolved.

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As seen in Figure 6, sample D-1 dissolved less silver than the other samples and thus did not precipitate silver before the reaction was stopped. Sample HN picked up the greatest amount of catalyst and began to precipitate at a higher concentration of peroxide than LN or D-3.

It is to be noted from the catalytic decomposition behavior of these four samples that a high starting activity is accompanied by a high catalyst erosion rate and a low starting activity is accompanied by a low catalyst loss. Examination of the physical properties listed in Figure 1 for these samples fails to indicate a gradation in any of the properties in the order of their activity and silver solution rate. On the other hand the chemical analyses shown in Figure 1 reveal that one ion, namely phosphate, does have the proper relationship to the decomposition behavior of these four production samples. Thus, the phosphate concentration is seen to be highest for sample HN, which has the highest starting activity and catalyst weight loss and lowest for D-1, which falls below the others in gas evolution and silver solution rates.

With this in view, it is of interest to see if this is a true effect of phosphate or merely a coincidence. The additive studies in the next section show that the phosphate concentration is indeed critical.

Catalytic Decomposition of Hydrogen Peroxide Samples Containing Additives

Although all four of the production samples used in the earlier studies with the static evaluator contained less than one milligram of phosphate per liter, their catalytic decomposition behaviors paralleled their phosphate contents. In addition, British investigators [reference (5)] have reported that the activity of peroxide is greatly increased by the addition of tin or aluminum. The undesirable behavior of some peroxide samples has also been attributed to nitrate ion [reference (1)]. Since these four substances are added to or readily picked up by hydrogen peroxide, it was desirable to determine their effects on decomposition characteristics in the static evaluator. This was readily accomplished by adding to a stock peroxide a dilute solution of a chemical containing the material of interest. The influence of some of the other common ions has also been studied in this additive program.

Phosphate -- The addition of phosphate ion to hydrogen peroxide greatly alters its behavior with a silver catalyst. That this type of behavior is dependent upon the concentration is shown in Figure 7. A small amount of phosphate, 0.5 mg./liters, increases the gas evolution rate of sample D-1 such that its activity

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corresponds closely to that of sample LN (compare with Figure 5). Increasing the phosphate content to 1.0 mg./liter steps up the starting activity to an even greater rate but the catalyst is apparently poisoned by the phosphate since the gas evolution rate decreases sharply after the first five minutes. At higher phosphate concentrations, 4.0 mg./liter or greater, the catalyst inhibition seems to occur earlier in a run so that the activity remains low throughout.

Examination of Figure 8 reveals that added phosphate not only alters the activity of a peroxide but also increases the solution rate of the catalyst. Here the relationship is more straightforward, the silver loss increasing with the phosphate content. The rising weight loss is small but steady up to 3 mg. phosphate/liter but jumps spectacularly above this value. In those runs in which the greater portion of the silver wire is dissolved, decomposition in the solution due to re-precipitation begins while the peroxide concentration is still above 75%.

With the other hydrogen peroxide production samples the critical effect of phosphate concentration appears to be the same although the full spectrum of phosphate content was not run. The increased starting activity and early catalyst inhibition at low concentrations and the progressive silver solution rate as phosphate increased were observed with samples HN, LN and D-3. No increased starting activity was observed with HN since its original activity already was very high. Sample D-3 exhibited an individualistic behavior in a concentration range of 2.0 to 2.5 mg./liter. Instead of being inhibited early in the run, the catalyst started decomposition at a high rate and maintained such a rate until the run was stopped as silver began to precipitate from the solution. It is thought that this property of sample D-3 may be due to its high aluminum content (Figure 1) but the relationship is not clear-cut at the present time.

The form in which phosphate is added to the peroxides is unimportant since 2 mg. phosphate/liter added as free phosphoric acid, monosodium phosphate, disodium phosphate, trisodium phosphate and sodium pyrophosphate gave similar decomposition phenomena. The apparent pH range with these additives at the given concentration varied from +0.65 for H_3PO_4 to +1.20 for Na_3PO_4 .

Tin -- Sodium stannate is one of the important stabilizers used for the prevention of decomposition of hydrogen peroxide in storage. However, some experimental work has indicated that tin in small concentrations can markedly increase the gas evolution rate on a silver catalyst in a laboratory evaluator [references (4) and (5)]. The present work has confirmed this higher

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activity in the initial part of a run. The curves reproduced in Figure 9 show that the starting activity for sample D-3 increased as the tin concentration was raised to 5 mg./liter. A sample containing 10 mg./liter gave essentially the same decomposition characteristics as one with 5 mg./liter. In addition to the increased starting activity with added tin, inhibition was observed in the later stages of reaction. This poisoning effect is not as pronounced as that found for phosphate ion but, as Figure 9 implies, its importance increases with the tin content.

Tin resembles phosphate in another undesirable aspect, namely, it increases the rate of solution of the silver catalyst in the peroxide. Here again the influence of tin is less than that of phosphate but it still is quite significant. In the static evaluator the silver loss was doubled for samples D-3 and D-1 when 5 mg./liter of tin was present. The catalyst loss for these two samples with varying amounts of added tin was essentially the same as shown in Figure 2.

In addition to the above, sample D-1, upon the addition of tin, also exhibited other phenomena similar to those already shown by sample D-3. Although at low concentrations the tin was not as effective in increasing the starting activity of sample D-1, the two behaved essentially the same when they contained 5 mg./liter. The poisoning action of tin on the silver catalyst was also evident. Sodium stannate had been added to sample D-1 as a stabilizer by the manufacturer and their analysis showed 3 mg./liter of tin at the time of shipment. It appears that the tin had been lost or at least converted into an ineffective form on storage in the aluminum drums. No runs with added tin were made with samples HN and LN.

Aluminum -- Storage and handling equipment for 90% hydrogen peroxide is usually fabricated from aluminum or its alloys. For this reason, the evaluation of the effect of aluminum on catalytic decomposition phenomena is necessary. It has been reported that aluminum in peroxide increases the starting and overall activity in decomposition on a silver catalyst [reference (5)]. That this is the case can be seen in Figure 10 which shows the gas evolution versus time curve for sample D-1 with added aluminum. The maximum effect seems to be reached at a concentration of about 1 mg./liter, little additional activity being observed when the aluminum content is raised to 2 mg./liter. Even smaller amounts of aluminum were sufficient to elevate sample D-3 to this maximum activity. The behavior of D-3 with 0.5 mg./liter of added aluminum was almost identical with that for D-1 plus 1.0 mg./liter. Aluminum is considerably more effective than tin in influencing the activity of peroxide. Thus 5 mg./liter of tin gave about the same starting activity as 1 mg./liter of aluminum when added to sample D-1. This would be reasonable if both were active on a molar basis instead of a weight basis since the atomic weight of tin is over three times that of aluminum.

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Aluminum does not show an increase in the rate of silver solution accompanying the increased activity in the decomposition of hydrogen peroxide. The catalyst weight losses for runs involving samples with added aluminum up to 2.0 mg./liter are listed in Figure 3. The differences are insignificant and probably within the reproducibility of the experiments. Samples D-1 and D-3 exhibited similar behavior in this respect.

As received, sample D-3 contained over 1.5 mg./liter of aluminum, a value several times that found for the other peroxides catalogued in Figure 1. This amount was found in the additive studies above to be sufficient to give a peroxide sample its maximum activity with a silver catalyst when the run was made immediately after aluminum addition. It is thus apparent that the aluminum originally present in sample D-3 was in a different form when introduced into the sample or else was converted by extended contact with high strength peroxide, heat or other agent into an ineffective form. Most of the aluminum additive program described in the present work utilized aluminum nitrate solution but similar results were obtained with aluminum chloride solution. It is concluded that the anion is not participating in the decomposition and that the entire alteration in behavior is due to the aluminum itself.

Nitrate -- Recent views have held that nitrate was partly responsible for high silver solution rates during catalytic decomposition of peroxide [reference (1)]. Early work in the present study seemed to corroborate this viewpoint since two runs with hydrogen peroxide containing five and ten mg./liter of nitric acid exhibited high silver losses. In addition, sodium nitrate appeared to have an inhibiting effect at 13 mg./liter and above. This work has been repeated many times but in no case has this behavior been duplicated. It is now felt that nitrate does not affect the decomposition in any way and that the unusual runs resulted from phosphate contamination which has been found to occur even when using extreme care.

Other Ions -- Sulfuric acid and hydrochloric acid can be present in the peroxide sample up to at least 10 mg./liter without altering the decomposition. This is likewise true of ammonium nitrate in a concentration of 30 mg./liter. Free ammonia, 6.5 mg./liter, has a slight inhibiting effect although it appears to be different from that of phosphate. It may be that this is merely a pH effect, since the apparent pH was shifted from 0.85 to 1.85 by the addition of the ammonia.

Catalytic Decomposition of Hydrogen Peroxide Samples Containing Two Additives

The work described previously has shown that the catalytic decomposition behavior of hydrogen peroxide can be greatly altered

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by the addition of certain additives. These materials, which are effective in very small concentrations, may afford desirable or undesirable properties. It is important to see if two additives with good features complement or nullify each other and if a good additive can cancel out the influence of a detrimental additive. For instance, will a combination of tin and aluminum give a higher starting activity for peroxide than either used separately or will the activity revert to that of the stock solution. The brief studies described below indicate that interaction can occur between mixtures of additives but more extensive work will be required to present a sharp picture.

Tin plus Phosphate -- A single run with sample D-3 containing 1.0 mg. each of tin and phosphate was made. The starting activity with this dual additive was greater than the initial gas evolution rate with either alone. The poisoning effect was slight but similar to that found for 1 mg./liter of tin in sample D-3. It is seen in Figure 4 that the catalyst loss was magnified by the simultaneous introduction of tin and phosphate. Thus the silver loss with both present was 18.4 mg., whereas that with tin was 12.0 mg. and that with phosphate was 15.2. Although it would be unwise to draw conclusions from a single run, further study of this combination should be worthwhile.

Phosphate and Aluminum -- A very high starting activity has been obtained for sample D-3 containing 0.5 mg./liter of aluminum. When 1.0 mg./liter of phosphate is also added to D-3, this activity is decreased considerably but still exhibits an activity comparable to D-3 containing about 0.35 mg./liter of aluminum. With 1.0 mg./liter of each of these ions, the behavior approximates a run with 0.4 mg./liter of aluminum in sample D-3. The limited data presented in Figure 4 for this pair of additives indicates that aluminum cuts down the effect of phosphate on silver solution rate. It should be noticed that this influence is greater at the higher aluminum concentration. The results of the two runs cited here point to an interaction between aluminum and phosphate which appears to reduce the effective concentration of both. A possible explanation for this behavior might be the formation of aluminum phosphate, a salt which has a low solubility in water.

Tin and Aluminum -- Beard and Smith [reference (5)] found that both tin and aluminum increased the activity of a silver catalyst in redistilled hydrogen peroxide, but observed only a slight increase when both were added to the same sample. It was of interest to see if a similar type of behavior would occur with production samples. In runs with peroxides containing both added tin and aluminum, the former was added to give a weight concentration four to five times that of the latter since it was shown in the studies above that aluminum was a more effective additive than tin.

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The tin retains most of its influence on the peroxide decomposition when present in conjunction with aluminum. The starting activity is usually enhanced slightly and inhibition at later stages of the reaction is still observed although the catalyst weight loss is reduced. Sample D-3, to which 1.0 mg./liter of tin and 0.25 mg./liter of aluminum had been added, started slightly faster than and generally paralleled the gas evolution versus time curve shown in Figure 9 for sample D-3 plus 1.0 mg./liter of tin alone. Comparison of data in Figure 4 indicates the silver loss observed with tin alone was reduced slightly when aluminum was included in the sample. This reduction was spectacular for sample D-1 plus 5.0 mg./liter of tin. The silver loss decreased from 23.5 mg. to 15.1 mg. as the result of the addition of 1.0 mg./liter of aluminum to this sample.

It appears that the effective concentration of tin may be reduced slightly by the addition of aluminum. This is not consistent with the British work cited above but it is possible that they were using aged samples. A sample containing both tin and aluminum exhibited lower starting activity and lower catalyst loss when allowed to stand five days prior to use.

Discussion -- The experimental results described in this report emphasize the critical effect of small amounts of foreign materials on the catalytic decomposition of hydrogen peroxide on a silver catalyst. While these laboratory studies are made under mild conditions it is probable that some of the difficulties encountered in decomposition units at high temperatures and pressures can be traced to the presence or absence of phosphate, tin or aluminum.

The erosion of silver catalyst and associated plugging of the catalyst bed can probably be attributed to phosphate although tin may be the culprit in some samples. In addition to plugging it has been speculated that a pressure build-up could also result from too high a catalytic activity as well as too low an activity. In the former case, decomposition in the fore part of the bed would result in a longer restricted path for the gas formed to traverse and a subsequent increased pressure differential through the catalyst bed. If this were true, a redesigned bed of shorter length or smaller surface area should solve the problem. Problems due to high activity might also be resolved chemically by elimination of activating materials such as tin and aluminum. Beard and Smith have proposed that low activity of a catalyst in high test peroxide results from a vapor binding phenomena which blocks the access of liquid peroxide to the catalyst [reference (5)]. In a wire screen catalyst bed, such an envelope of vapor surrounding each wire would reduce the area available for fluid flow and conceivably build-up a considerable pressure head. Colloidal materials such as alumina and stannate overcome this problem in laboratory apparatuses.

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If the activity of peroxide in a high pressure decomposer can be altered as simply as it is in a laboratory apparatus, the part played by activity in the pressure build-up can be evaluated. This question can be answered only by a similar study of additives in a high performance decomposer. It is conceivable that the availability of a uniform, high activity peroxide would permit a reduction in catalyst weight and size.

Although the decomposition properties of hydrogen peroxide can be drastically changed by addition of small amounts of material, there is evidence that these substances do not retain their activity when stored for a period of time. This is particularly true of tin and this study gives the first indication that aluminum may also lose its influence on the catalytic decomposition of peroxide. The behavior of hydrogen peroxide containing two or more of the critical materials - tin, aluminum and phosphorus - is undoubtedly complex and probably depends upon the concentration of both additives as well as length of storage and the material of which the container is made.

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Figure 1

Physical Properties and Chemical Analyses of
Production Samples of Hydrogen Peroxide

	<u>D-1</u>	<u>D-3</u>	<u>LN</u>	<u>HN</u>
Apparent pH	+ 1.0	+ 0.8	+0.4	- 0.6
Conductivity, mhos X 10 ⁻⁶	11.7	10.3	9.7	20.6
Phosphate, mg./liter	0.1	0.5	0.6	0.9
Aluminum, mg./liter	0.2	1.6	0.3	0.2
Nitrate, mg./liter	2.4	2.5	2.5	10.4
Sulfate, mg./liter	7.9	5.4	5.5	5.0
Tin, mg./liter*	3.0	< 0.01	---	---

- * Tin analysis for D-1 reported by manufacturer.
Tin analysis for D-3 by spectrographic analysis of an
evaporation residue.
LN and HN were not analyzed for tin but none had been
added by the manufacturer.

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Figure 2

Catalyst Weight Loss in Decomposition
of Hydrogen Peroxide with Added Tin

Tin Concentration* (mg./liter)	Catalyst Loss (mg.)	
	D-1	D-3
0.0	10.9	10.0
1.0	13.2	12.0
2.0	15.7	14.8
3.5	15.6	15.3
5.0	23.5	22.1
10.0	21.6	21.5

* Tin added as 0.14% $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$.

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Figure 3

Catalyst Weight Loss in Decomposition
of Hydrogen Peroxide with Added Aluminum

Aluminum Concentration* (mg./liter)	Catalyst Loss (mg.)	
	D-1	D-3
0.0	10.7	9.7
0.25	--	10.1
0.35	--	11.0
0.40	--	10.0
0.50	9.8	11.3
1.00	10.0	11.0
1.40	10.7	--
2.00	10.8	--

* Aluminum added as 0.17% $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$.

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Figure 4

Catalyst Weight Loss in Decomposition
of Sample D-3 with Additives

<u>Additive</u>	<u>Additive Concentration (mg./l.)</u>			<u>Catalyst Loss (mg.)</u>
	<u>Sn</u>	<u>PO₄</u>	<u>Al</u>	
None	--	--	--	9.5
Sn	1.0	--	--	12.0
PO ₄	--	1.0	--	15.2
Al	--	--	1.0	11.0
Sn + PO ₄	1.0	1.0	--	18.4
PO ₄ + Al	--	1.0	0.5	12.9
	--	1.0	1.0	11.9
Sn + Al	1.0	--	0.25	10.8

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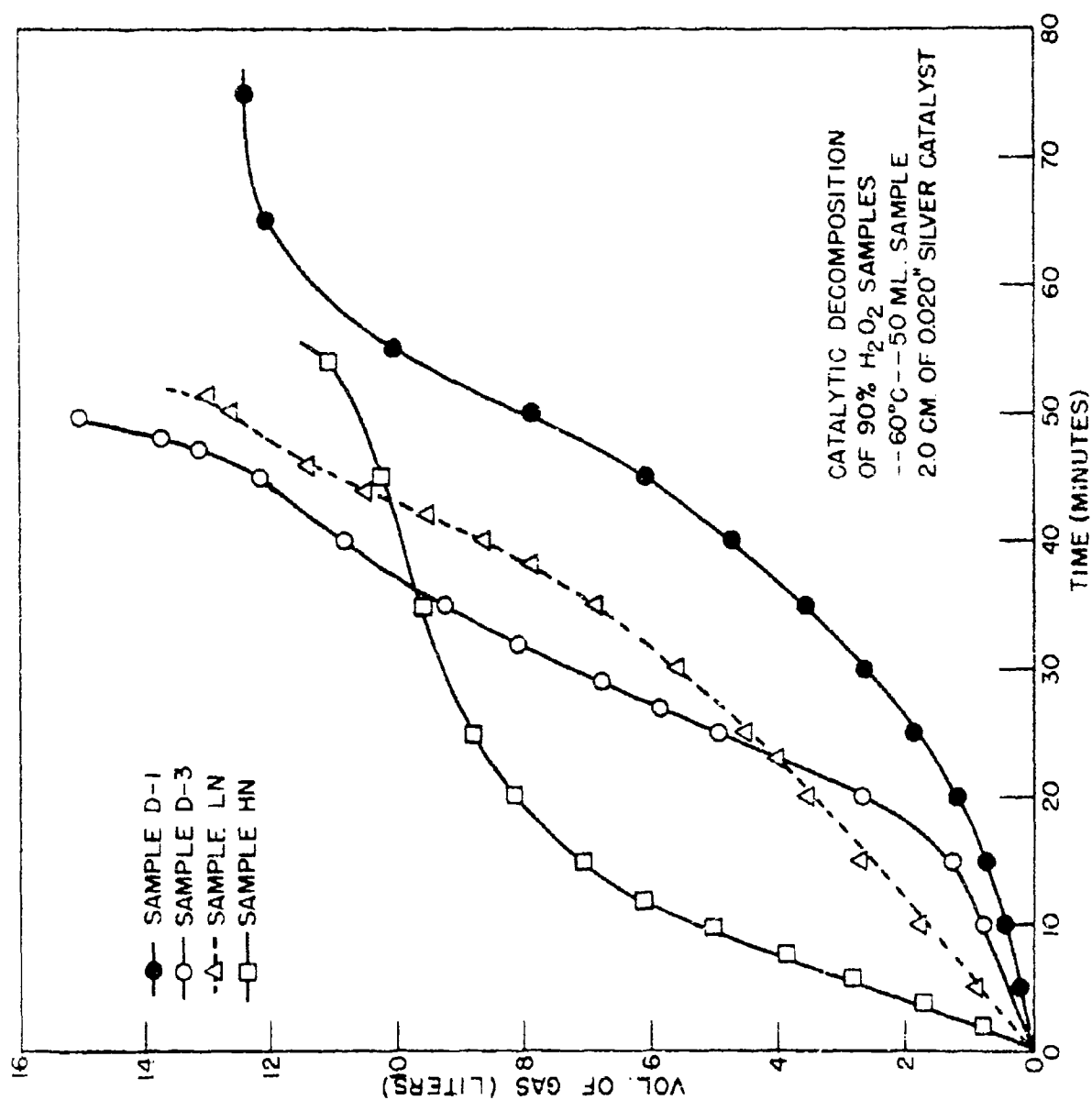
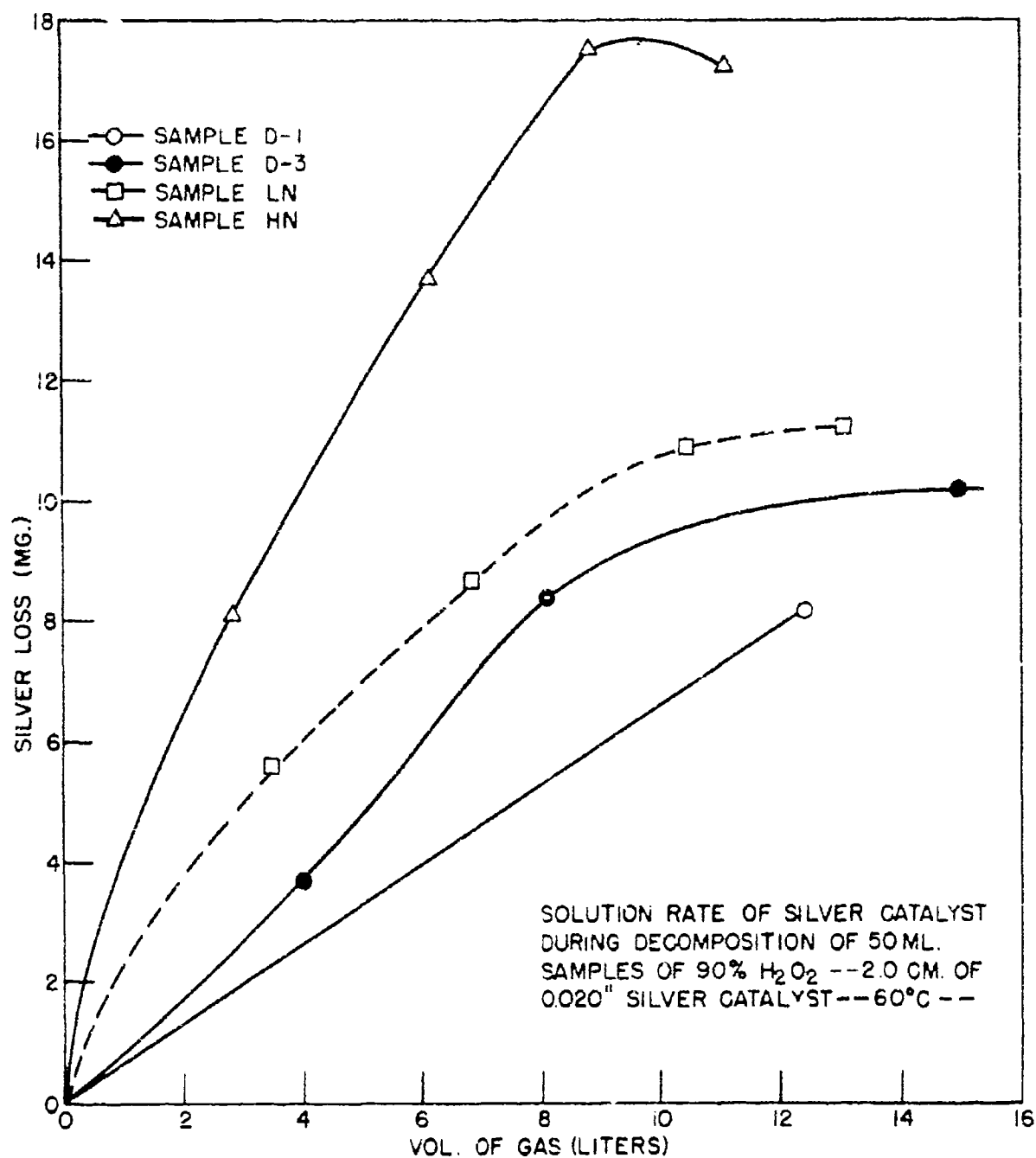


Figure 5

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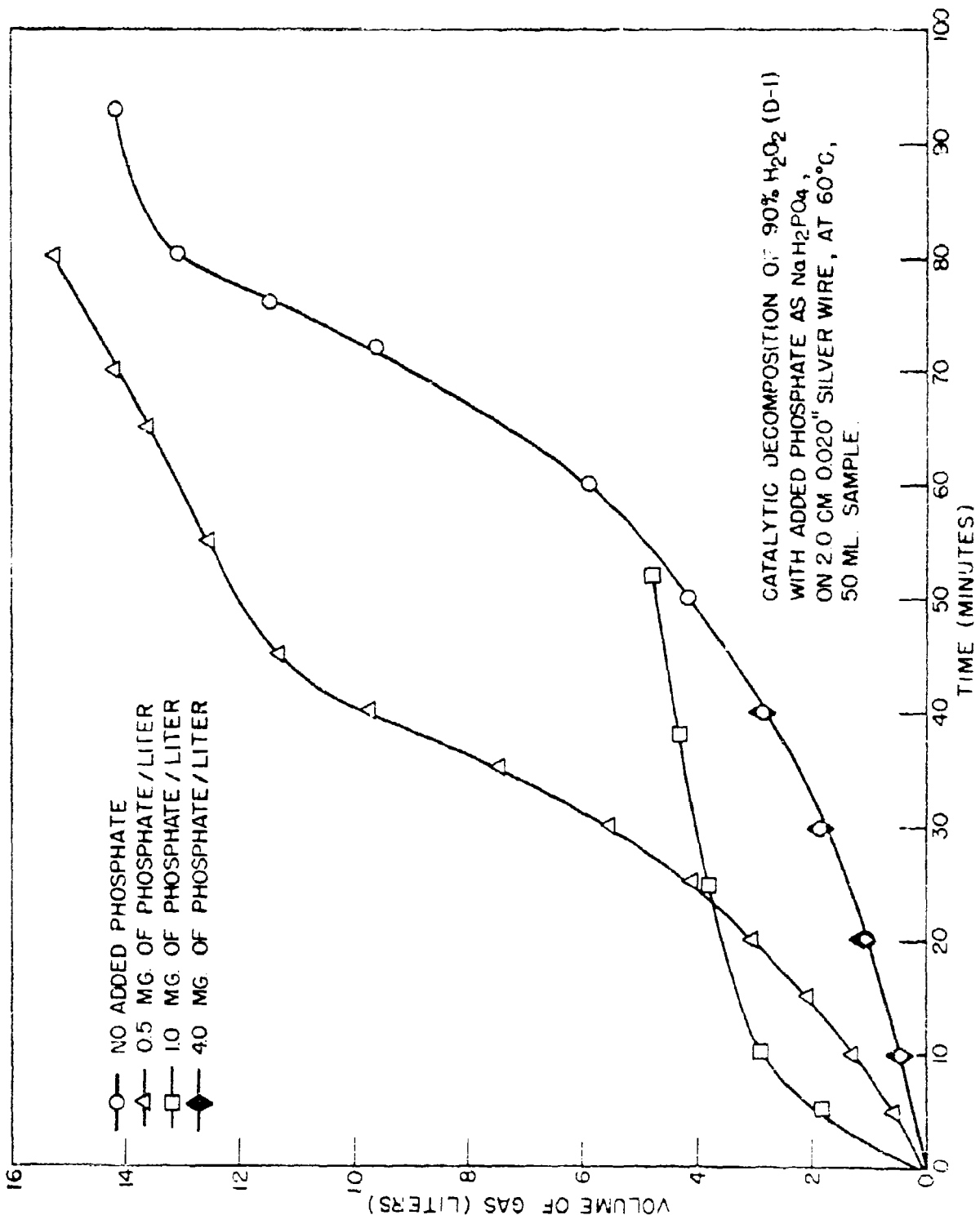
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Figure 6

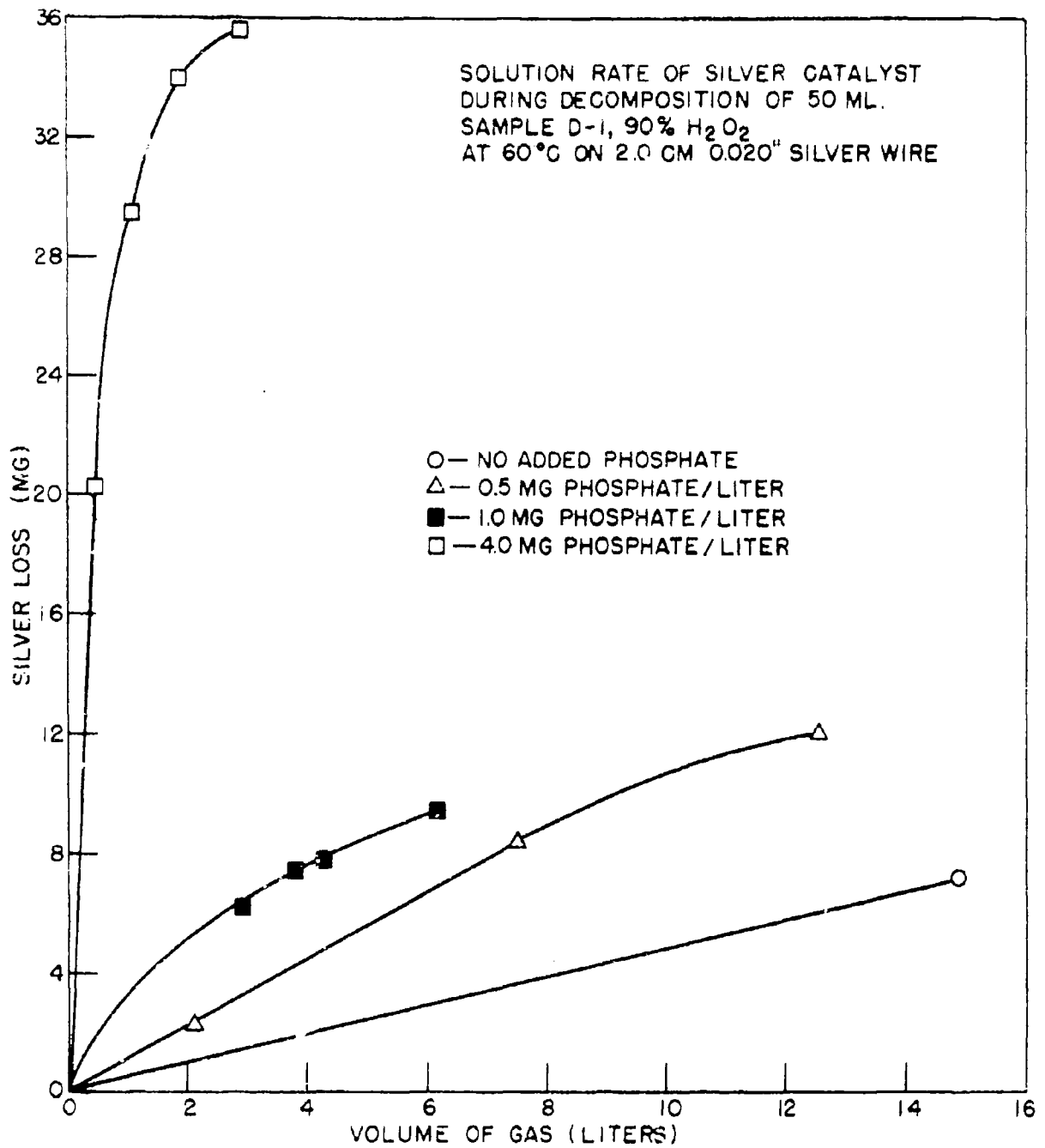
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Figure 7

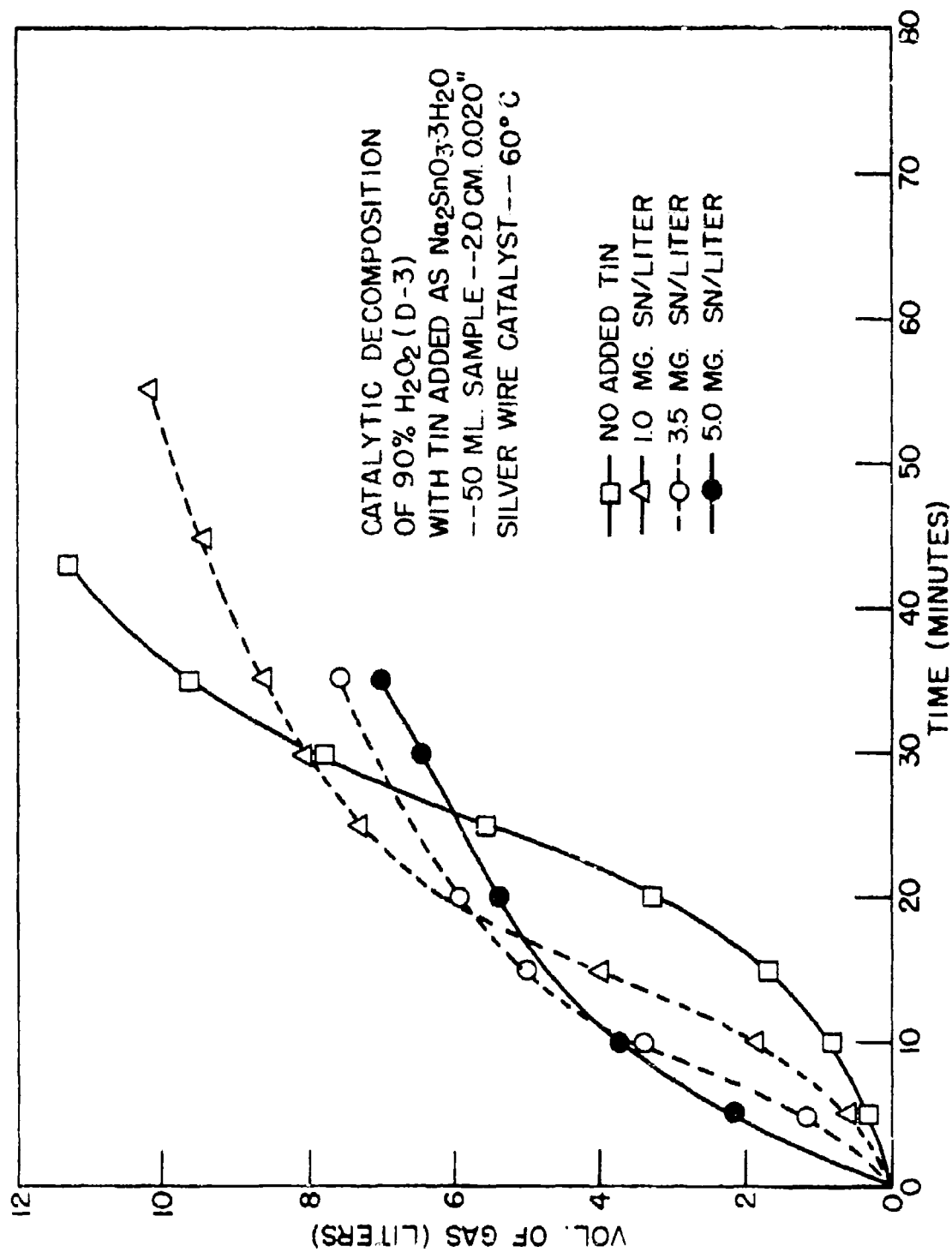
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Figure 8

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Figure 9

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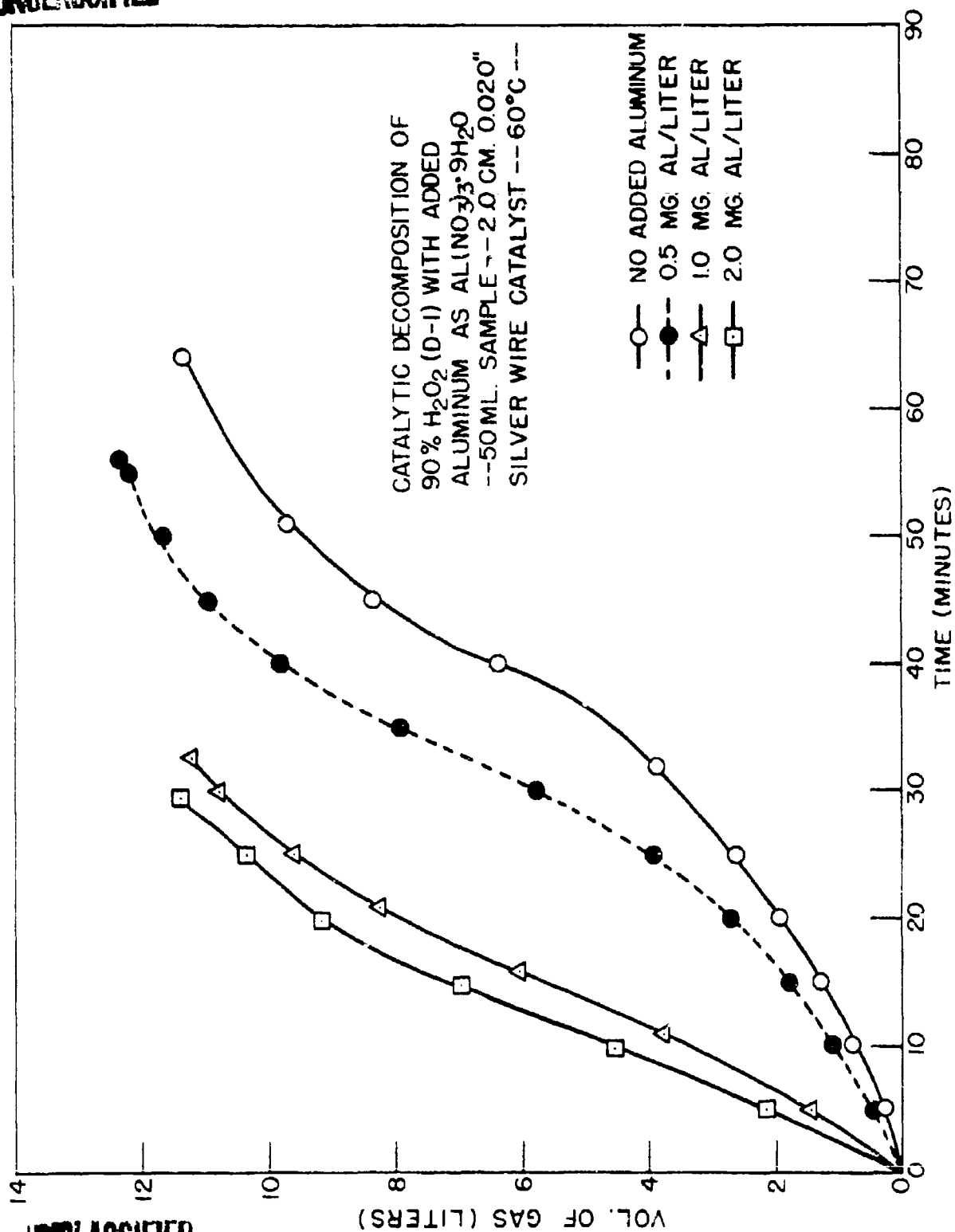


Figure 10

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